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Characterization, I. R. Spectrophotometric, GC- Ms. and Free Fatty Acid Profile Analysis of Extracted Oil from Almond Tamilanida Catappa Tree Fruits Seed

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Abstract

Almond catappa tree is one of the C3 plant in Nigeria tropics, usually planted as an umbrella tree and as a source of fruits. Therefore, the need to investigate and characterized the extracted oil from the fruits seeds, using standard methods. The physicochemical parameters results reveal the Free fatty acid value as 8.46±0.04%, acid value: 14.025±0.02mg/KOH, saponification value: 446.57±0.01mg/g, peroxide value: 180.00±0.02meg/kg, Iodine value: 1.147±0.01gI_2/100g, Refractive index: 1.426±0.01, Viscosity: Density: 0.824±0.01g/$cm^3$and has a light yellow color. Peroxide value and saponification value, was the only high value observed among the physic chemical parameters. The infra-red spectrum of the sample reveal nine major peaks: they are 3005.9cm^{-1}, 2922.64cm^{-1}, 2853.33cm^{-1}, 1744.57cm^{-1}, 1458.15cm^{-1}, 1377.36cm^{-1}, 1159.31cm^{-1}, 1097.57cm^{-1}, 721.73cm^{-1} and The results of the poly aromatic hydrocarbon compound present confirm the use of the oil as a petroleum product because of the double and condense rings present, which are: naphthalene, fluorene, acetylnaphthalene, anthracene, aenaphthalene, fluoroanthrene, chyrene, benzylanthracene, benzol(b)fluoroanthrene and pyrene. It also contain sixteen fatty acids, with the oleic acid: 62.0737%, linoleic acid: 18.9768%, palmitic acid: 12.5099%, stearic acid: 4.8367% and the rest were bellow 1%.

In conclusion, the oil does not compared favorably with other vegetable oils properties, and to ascertain its quality, also, the present of benzaldehyde at 3005.9cm^{-1} as C-H stretch for aromatic benzene ring, peaks at 2922.63cm^{-1} and 2853.33cm^{-1} as aliphatic C—H stretching of an aldehyde group, peaks at 1744.57cm^{-1} is due to carbonyl aromatic aldehyde group and mono substituted benzene at 721.73cm^{-1} and the presence of poly aromatic hydrocarbon indicated its property as one of the oils that can be used in processing petroleum.

Keywords: Characterization, Oil Contents, I. R. and GC- Ms Analysis

Introduction

Catappa almond tree is one of the tree generally grown as decorated tree (umbrella Tree) in most Nigerian environments especially in University campuses both as a fruits sources and for research purposes because of its deciduous character among other trees and its medicinal *use of the fruits. O*ther tree in this categories include flamboyant tree, It normally fruits more than three times in a year and when properly planted where there adequate water sources it fruits profusely normally the fruits were arranged in whorled of 4—8 per fruits nodes stands. It is a tropical deciduous tree. It usually grow between 30m -- 50m foot tall tree which form a symmetrical upright in growth with horizontal branches reaching 35ft in width. The branches arranged in obvious pattern giving the tree a pagoda like shape. The large 12 inch long and 6 inch, fruits include beautiful shape of red, yellow and purple, and leaves before dropping by a process called Thigmomorphogenesis. The phenomenon of leaves dropping usually takes place when there is loss of nutrients or element like Ca^{2+} leading to tail end of the leaves region, curve and the leaves margin changes to brown and latter drops. The dropping and replacement of leaves at the bud of the tree is control by hormones like auxin and abscidic acid (ABA). The leaves were quickly replaced by new growth as a results, the tree is bared for a short period of time [about 2 ½ weeks] but the leaves starts growth almost immediately it drop. The inconspicuous greenish white spring out, blossom appear in six inch long terminal, cluster and are follow by the edible fruits

between 2 or 4 -to- 8in number. These drupe of about 2 - 5 inches long and mature from green to yellow or red during summer. The outside husk is curkey fibre with an inner greenish flesh. (Edward 1994). The fruits when mature properly and dried has a hardy kernel that enclose the seed, which is whitish, with brown seed coat usually one or two.
Moreover, the tree seed oils contain benzaldehyde, that makes its oil as one of the oils used in processing petroleum as reported by [15]. and other Plant biology books. The tree dried trunk is also use in the formulation of (NPK) fertilizer for soil improvement as one of the deciduous tree together with other components and also the fruits has medicinal properties due to the presence of phytochemical.
Justification the various uses and application of this tree both as fruits source in terms of vitamin C, phytochemicals and as fertilizer because of its deciduous properties, and as a source of potash, finally the seed oil as source of petroleum product due to its constituents and present of benzaldehyde all these necessitate the study of the fruits seed oil, characterization, infra red spectrophotometric and gas chromatography—Mass spectrometry analysis in other to actualize the potentials.

Fig 1: Catappa Fruits

Exracted Oil sample

Prepared Seeds for processing

Experimental
Material and Method
Sample preparation: The mature fruits of almond catappa tree was picked at the surroundings after being drop either by wind or natural occurrence. The fruits were taking to the laboratory for air drying and later removed the seed inside with the use knife and a table vice, by placing the scraped fruits between the vice jaw and screwed up until the hearing of pop sound, then remove the seed. The seeds was immediately dried in an oven M300 series at 105°C for 1—2 hr. and allowed to cool, then blended with Kenwood blender (M201) fission and store in dried clean PVC plastic.
Methods: The sample was characterized for Free fatty acid, peroxide value, acid value, iodine value content, saponification value, refractive index and viscosity together with infra red, and Gc – Ms analysis. Using standard method and the chemical used were BDH standard chemical. All the experiment was carried out in triplicate.

Determination of Free fatty acids
5g of oil sample was weight into a clean 250ml volumetric conical flask, 50ml of neutral alcohol was added to the content and was titrated with 0.1M sodium hydroxide. Using phenolphthalein indicator.

$$\% \text{ free fatty acid; } \frac{\text{Titre value x N xF x 100}}{\text{Weight of sample}}$$

N: Normality of NaoH. F: equivalent weight of free fatty acid. Conversion factor for oleic acid =282 The experiment was carried out in triplicate.
Acid Value: 10g of well mixed oil sample was weighed into a clean volumetric conical flask, 25ml of diethyl ether and 25ml of ethanol were added and three drops of phenolphthalein was added to the mixture. The mixture was titrated with 0.1M KOH to the end point with consistent shaking for which a pink color was observed and the KOH volume used noted [10, 7].

$$\text{Acid Value: } \frac{\text{Titre value x N x 56.1}}{\text{Sample weight}}$$

N: Normality of KOH 56.1: molecular weight of KOH. The experiment was carried out in triplicate.
Peroxide Value: 5g of sample was weigh into a clean volumetric conical flask 30ml of acetic acid – chloroform solution, the content was shaken until the oil dissolved, 0.5ml of saturated potassium iodide was added using mohr pipette, the solution was left to stand for one minute with occasional shaken and then 30ml of distilled water was added, shaking then titrated with 0.1N sodium thiosulphate, adding gradually with constant shaking until tallow color has almost disappear, 0.5ml of starch indicator solution and continue titration with shaking until the end point, when all the iodine has librated from chloroform layer. At this period the blue black appear. The blank titration was also conducted. All experiment was conducted three times and at different stipulated temperature.

$$\frac{\text{(S --- B) x N x 1000}}{\text{Weight of sample}}$$

S = Titration blank, B = sample titration, N = normality of thiosulphate. The experiment was carried out in triplicate.

Saponification Value: 5g of the oil sample was dissolved in 25ml of 10% alcoholic KOH. The solution was refluxed for 40 minutes to saponify the oil. The unreacted KOH was back titrated while hot with standardized 1.0M HCL, using phenolphthalein indicator [10].
S = sample titre value,
56.1 = The molecular weight of KOH. The experiment was carried out in triplicate.

$$\text{Calculation: } \frac{\text{Titre value x Molar mass of KOH}}{a}$$

a = weight of the oil sample.

Determination of iodine value
5g of sample was weight into a volumetric flask containing 20ml carbon tetrachloride and 25ml of wijs solution and shaken for complete mixing. This content was stored in the dark for 30minute at 25ºC latter titrated against 0.1N sodium thiosulphate after adding 20ml of potassium iodide solution.

$$\text{Iodine value: } \frac{(B - T) \times N \times 12.69}{\text{Weight of sample}}$$

B = blank titration, T = titration value, N = Normality of thiosulphate.
Viscosity: This was carried out with the use of viscosity meter. The experiment was carried out in triplicate.
Refractive Index: The oil sample refractive index was carried out with Abe refractometer instrument. Model: KenttN23ey.
Infra Red Spectrum analysis of the extracted oil: This was carried out at the University of Ibadan multidisciplinary Research Laboratory. Ibadan.
Aromatic Compound Present: This was carried out using Gc—Ms Agilent. Technologies with auto sampler.
GC – Ms analysis on the free fatty acid profile of the extracted oil: The processed fatty acid methyl ester was analysed at the Central Research Laboratory Federal University of Technology Akure.
Reagents: wijs solution 13g of iodine was dissolved in a litre of glacial acetic acid, heated solely for easy dissolution and cool before use.
Potassium iodide solution: 15g of KI dissolved in one liter of distilled water. Starch indicator: 10g of soluble starch in small cold water and latter add one liter of boiled water, stirred rapidly and cool.
Sodium thiosulphate: 24.82g of sodium thiosulphate and 3,8g of borax and make up to mark to one litre. The addition of borax is to prevent bacterial deterioration.

Statistical Analysis
Data were subjected to analysis of variance (ANOVA) as describe by Ihekoroye and Ngoddy (1985) Means were different, separated by least significant different (LSD) test, significant was accepted at $p < 0.05$.

Results, Discussion and Conclusion

Table 1: Results of the Physicochemical Parameters of the extracted oil

Parameters	Mean Value
Free Fatty Acid % as oleic acid	8.46±0.04
Acid value mg/KOH	14.025±0.02
Saponification value mg/KOH	446.57±0.01
Peroxide value meg/100g	180.00±0.01
Iodine value gI_2/100g	1.167±0.01
Refractive index	1.42±0.01
Viscosity mpa/S	1.414±0.01
Density/ specific gravity	0.824±0.01
Color	Light yellow

The above table 1 reveal the free fatty acid value of the oil sample to be 8.46±0.04. The degree of edibility of a fat is generally considered to be inversely proportional to the total amount of the free fatty acid of the oil [3, 11]. The value is lower to that value reported for bottle: 21.40±0.02% and ornamental:19.50±0.01% gourd seeds oil but higher to that of ash gourd:2,68±0.02% seeds oil by [5] and also lower to that reported for palm oil 29.00±0.05% by [14]. from Okitipupa located in Ondo state, and value reported for valential peanut seed oil: 66.55±0.06% by [6]. The acid value observed: 14.025±0.02mg/KOH for the oil sample was higher than the value reported for oil extract from fermented seed of c. populnea.2.44±0.50 mg KOH/g by Mathew *et al*. 2020. It is also higher than the value reported for Tephrosia vogelii seed oil: 1.647mgKOH by [4], and also higher than the value reported for castor oil seed oil undehydrated: 3.83mgKOH/g of oil by [8]. The peroxide value observed: 180.00±0.02 meg/100g it is more higher than the value reported for moringa (amichi) seed oil: 1.14meg/kg by [12] and also higher than values reported for palm kernel oil at different temperature: at 100ºC at 40minutes: 3.39±0.01meg/kg, at 150ºC at 40minutes: 5.56±0.02meg/kg, at 200ºC at 40munites: 7.03±0.02meg/kg as reported by [11], and that peroxide increases as temperature increases and the lower the viscosity as reported by [11]. The iodine value observed was: 1.167±0.01gI_2/100g, It is more lower than the value reported for moringa (amichi): 123.0mg/g by [12], and also lower than the value reported for castor oil undehydrated: 83.44gI_2/100g oil, as reported by [8]. It was also lower to the value reported for Coconut seed oil (*Coco Nucifera*): 7.20±0.007bI_2/100g as reported by [3]. The iodine value indicate the level of unsaturated fats present in the oil sample. The refractive index observed value: 1.426±0.01, this value was slightly lower than the value reported for castor oil undehydrated: 1.415 and lower to that reported for irivingia gabonensis seed oil: 1.4425±0.41 by [8, 12]. The saponification value observed was 446.57±0.01mg/g and is higher than value reported for moringa Oleifera seed oil (Amawbia type): 168.01 mg/g by [12]. The density of the extracted oil observed value was 0.824±0.01g/cm^3, this value is lower to the value reported for castor seed oil

unhydrated: 0.962 as reported by [8]. The density value is also higher than the value reported for moringa oleifera (amawbia type): 0.75g/cm^3 by (Muoka and Ibeh 2018). While the color was light yellow.

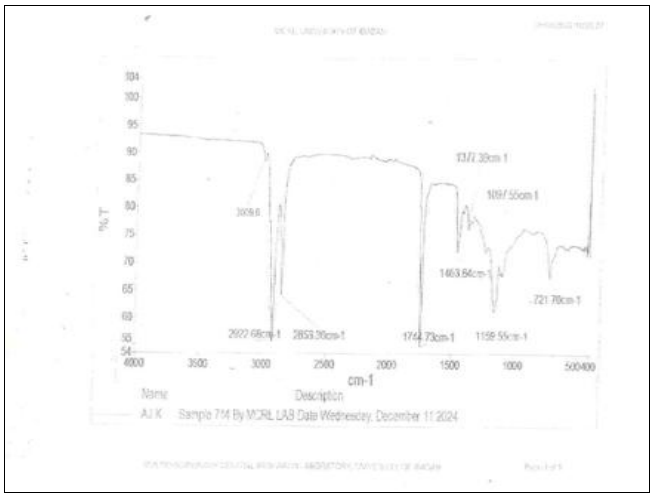
The viscosity value observed was 1.414±0.01mPa, the value is lower to the value reported for bottle gourd: 31.60±0.02 mpa by [6]. It was also lower to the value reported for ipomoea seed oil: 8.61 (cst) by [11].

Table 2: Poly AromaticCompound Chemical Constituents of the oil sample

S. No	Name of Compound	Amount %
1	Naphthalene	5.819
2	Acetylnaphthalene	7.548
3	Aenaphthalene	8.200
4	Flourene	8.641
5	Phenanthrene	9.642
6	Anthracene	10.174
7	Fluoanthrene	11.233
8	Pyrene	11.233
9	Benzyl anthracene	12.611
10	Chryene	12.611
11	Benzol(b)fluoranthrene	13.803
12	Benzol (k) fluoranthrene	14.055

The above table 2 indicate the results table for the aromatic chemical constituents of the extracted oil.

They are all poly Aromatic hydrocarbon compounds which are Naphthalene 5.819%, Acetylnaphthalene, 7.548%, Aenaphthalene 8.100%, Fluornene 8.641%, Phenanthrene 9.642%, Anthracene 10.174%, Fluoanthrene 10.966%, Pyrene 11.233%, Benzyl anthracene 12.611%, Chyrene 12.611%,Benzol (b) fluoroanthrene 13.803% and benzol (k) fluoroanthrene 14.033%. This compounds are use in petroleum processing. The above mention chemical compounds are the major component of petroleum and it express how important this seeds oil to refining industry and the need for mass cultivation. Since the seed oil contain polyaromatic compound then the whole fruits is likely to contain the polyaromatic compound also.



The above Infra-red spectrum for the sample indicate nine major peaks which are: 3005.9cm^{-1}, 2922.63cm^{-1}, 2853.33cm^{-1}, 1744.57cm^{-1}, 1458.15cm^{-1}, 1377.36cm-1, 1159.31cm^{-1}, 1097.57cm^{-1} and 721.73cm^{-1}. The aromatic benzene ring peaks at 3005.9cm^{-1} as C-H stretching and 2922.63cm^{-1} and 2853.63cm^{-1} peaks that represent aliphatic C—H stretching of an aldehyde group and CH_2 assymmetric and symmetric vibration at 2922.63cm^{-1} and 2853.33cm^{-1} and CH_3 group with C-H deformation vibration modes at the asymmetric vibration at 1458.15cm^{-1} peaks why peak at 1744.57cm^{-1} represent stretching for carbonyl C= O for saturated aromatic aldehyde group and peak at 1458.15cm^{-1} confirm the compound to be aromatic in nature (Arene) in addition, peaks at 2853.33cm^{-1} indicate methyl C-H stretching vibration, and peak at 721.73cm^{-1} is due to C-H bending vibration of mono substituted benzene ring. All peaks values were analysed with the standard data value.

Table 3: Results of Free Fatty Acid Profile of Extracted oil Sample

Parameters	Normal amount present %
Caylic acid methyl ester	0.001228
Capric acid methyl ester	0.000658
Lauric acid methyl ester	0.0011583
Myristic acid methyl ester	0.008505
Palmitic acid methyl ester	12.509912
Palmitoleic acid methyl ester	0.450776
Margaric acid methyl ester	0.012281
Stearic acid methyl ester	4.826784
Oleic acid methyl ester	62.073674
Linoleic acid methyl ester	18.996788
Linolenic acid methyl ester	0.173998
Arachidic acid metyyl ester	0.663987
Arachidonic acid metyl ester	0.013502
Behenic acid methyl ester	0.244129
Erucic acid methyl ester	0.016519
Lignoceric acid methyl ester	0.028221

Table 3 above reveal the presence of sixteen free fatty acid present in the analyzed sample. They are: Caprylic acid methyl ester, capric acid methyl ester, lauric acid metyk ester, myristic acid methyl ester, palmitic acid methyl ester, palmitoleic acid methyl ester, margaric acid methyl ester, stearic acids methyl ester, stearic acid methyl ester, Oleic acid methyl ester, Linoleic acid methyl ester, Linolenic acid methyl ester, Arachidic acid methyl ester, Arachidonic acid methyl ester, Behenic acid methyl ester, Eruicic acid methyl ester and Lignoceric acid methyl ester. The oleic acid methyl ester has the highest percentage amount present (62.073674%) followed by Linoleic acid methyl ester (18.976788%) and the lowest percentage with Capric acid methyl ester (0.000658%). This constituent is comparable with other oils but with different amount.

Conclusion

The analysis carried out on the extracted oil from tamilida catapa fruit seeds as it was observed from the different results reveal the oil as having multi property as oil with the present of polyaromatic hydrocarbon, benzaldehyde with sixteen free fatty acid methyl ester and as a fruit having phytochemicals presence in it, that makes some people eating it as edible fruit and now with its polyaromatic compound presence it will be good as one of the oil that can be use in processing petroleum.

Recommendation

If this oil is treated with other plant and material that has similar Poly aromatic hydrocarbon and used engine oil and used gear oil (they contain PAH, these, will increase and boost the poly aromatic compound present and it will be more effective for what Poly aromatic hydrocarbon are used for in industry. The waste generated from this plant fruits as a result of few people eating them can be use for the oil

production, even the whole crushed and extracted fruits was detected to contain poly-aromatic compound. And can be put to use more effectively. The pollution prevented act 1990 and Environmental Protection Agency (EPA) 1991 should be put in place considering resources conservation and recovery act for this useful plant.

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